

# PATENT ABSTRACTS OF JAPAN

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## (54) CERAMIC RESISTOR

### (57)Abstract:

**PURPOSE:** To obtain a resistor having small thermal variation by controlling the quantity of element of periodic table 4b group in aluminum nitride and lattice constant.

**CONSTITUTION:** An aluminum nitride film, on which a periodic table 4b group element is, excessively solidified on the surface of a substrate consisting of an aluminum nitride sintered body, is compounded by a CVD method. As a result, a ceramic resistor, containing 0.005 to 30atom% of the element of 4b group on the periodic table, on which the lattice constant of aluminum nitride is shifted by 0.003 to 0.030 Angstrom on a-axis and 0.004 to 0.080 Angstrom on c-axis, can be obtained. Besides, the volume intrinsic resistance at 25° C becomes 1013Ω/cm or smaller, and the volume intrinsic resistance becomes 1013 to 1011Ω/cm in the temperature range from room temperature to 300° C. Accordingly, the usefulness of the ceramic resistor is high when it is used for the electrostatic chuck of a semiconductor manufacturing device on which stabilized resistance is required for a wide temperature range.

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CLAIMS

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(57) [Claim(s)]

[Claim 1] the ceramic resistor which makes an alumimium nitride crystal phase a subject -- it is -- the inside of this resistor -- the [ periodic table ] -- the ceramic resistor characterized by the volume resistivity in 25 degrees C being 1013 or less ohm-cm while 4b group element is the value which was carried out and only 0.004-0.080A of lattice constants in said crystal phase shifted from the lattice constant of alumimium nitride single phase by 0.003-0.030A and the c-axis by the a-axis 0.005-30 atom % existence.

[Claim 2] The ceramic resistor according to claim 1 in which said resistor is formed by the chemistry vapor phase synthetic method.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the ceramic resistor which makes a subject the alumimium nitride suitable for a heater ingredient, the electrification removal ingredient in thermionic-tube enclosure tubing or semiconductor fabrication machines and equipment, the arm for wafer conveyance, the fixture for wafer handling, etc.

[0002]

[Description of the Prior Art] Conventionally, generally as an approach for adjusting the electric resistance of the insulating ceramics, adding a conductive ingredient and controlling resistance is performed to the insulating ceramics. For example, adding titanium nitride to an alumina and making electric resistance small is performed.

[0003] On the other hand, alumimium nitride is one sort of the non-oxidizing quality ceramics, the application as a structural material or high temperature materials is expected, and having the endurance which was excellent in recently also to the plasma-proof is reported. Therefore, the application as components in semiconductor fabrication machines and equipment, such as an electrostatic chuck, is taken into consideration in this alumimium nitride. However, it is this alumimium nitride itself and a high insulating material, and since it has the resistance of 10<sup>16</sup> or more ohm-cm also at a room temperature, the present condition is having not resulted in utilization.

[0004] The attempt which makes electric resistance small is performed also to such alumimium nitride. For example, adding conductive ingredients, such as aluminum, also to the insulating ceramics of alumimium nitride or boron nitride, and adjusting specific resistance is proposed by JP,56-4509,A. Moreover, in the thin film-like ceramics, making alumimium nitride distribute metal aluminum for example, and obtaining a thin film resistor with a small temperature coefficient of resistance is also proposed by JP,55-50364,B.

[0005]

[Problem(s) to be Solved by the Invention] Generally, although the volume resistivity value of an insulator tends to fall with temperature, in the case of alumimium nitride, it is in the inclination which decreases to 10<sup>11</sup> or less ohm-cm by 300 degrees C from 10<sup>16</sup> ohm-cm at a room temperature, for example. Therefore, since the actuation by which resistance was changed and stabilized was not obtained when using it from a room temperature to a 300-degree C elevated temperature, there were problems, like service temperature conditions have a limit.

[0006] Moreover, in the approach of controlling electric resistance, there were problems -- the property which the insulating ceramics originally has with the property of the conductive ingredient itself is spoiled -- by adding a conductive ingredient. For example, corrosion resistance and endurance were missing and the property of alumimium nitride deteriorated.

[0007]

[Means for Solving the Problem] The result to which, especially as for this invention person etc., electric resistance repeated examination from a viewpoint of the presentation and an organization as a ceramic resistor of 10<sup>13</sup> or less ohm-cm to the above-mentioned trouble, 0.005-30 atom % content of 4b group element group's element is carried out. for example, the inside of the insulator which uses as a principal

component the aluminum nitride formed by the chemistry vapor phase synthesis method -- the [ periodic table ] -- By making the element dissolve during an aluminum nitride crystal, and controlling the lattice constant of aluminum nitride in the specific range The volume resistivity of an insulating layer could adjust to the range of  $10^{13}$  or less ohm-cm, and it found out that the material property by which the temperature change was stabilized in the small large temperature region was acquired, and resulted in this invention.

[0008] namely, the ceramic resistor to which the ceramic resistor of this invention makes an aluminum nitride crystal phase a subject -- it is -- the inside of this resistor -- the [ periodic table ] -- 0.005-30 atom % existence, it carries out, and 4b group element is characterized by the volume resistivity in 25 degrees C being  $10^{13}$  or less ohm-cm, while the lattice constant in said crystal phase is in the value shifted only 0.004-0.080Å by 0.003-0.030Å and the c-axis by the a-axis.

[0009] Hereafter, this invention is explained in full detail. although the ceramic resistor in this invention is what makes aluminum nitride a subject -- the [ presentation top and periodic table ] -- 0.005-30 atom % content of 4b group element is done. the [ this / periodic table ] -- if the amount of 4b group elements is an important element for giving conductivity to aluminum nitride, desired resistance will not be obtained if there are few these amounts of elements than 0.005 atom %, but 30 atom % is exceeded -- other crystal phases -- generating -- being easy -- rheostatic control becomes difficult and it becomes easy to generate exfoliation and a crack in a thin film. the [ in addition, / periodic table ] -- 4b group elements are specifically C, Si, germanium, Sn, and Pb, and especially C and Si are desirable in respect of membrane formation nature.

[0010] moreover -- although this ceramic resistor is what makes an aluminum nitride crystal a subject on an organization -- the [ in this resistor / periodic table ] -- the [ which cannot dissolve during this crystal although some 4b group elements dissolve during an aluminum nitride crystal / periodic table ] -- 4b group element -- the [ periodic table ] -- it comes out comparatively, and there are crystal phases, such as 4b group element group's nitride, also when [ 20 or less % of the weight of ] it exists. moreover, an aluminum nitride crystal -- the [ periodic table ] -- being in the range of the value which only 0.004-0.080Å of lattice constants shifted from the lattice constant of aluminum nitride greatly or small by 0.003-0.030Å of a-axes, and the c-axis by dissolution of 4b group element, the lattice constant (3.120Å of a-axes, 4.994Å of c-axes) of the crystal which consists of an aluminum nitride simple substance has a clearly different lattice constant.

[0011] The ceramic resistor of this invention has the volume resistivity of  $10^{13}$  or less ohm-cm in 25 degrees C by the above-mentioned configuration, and the lower limit is about 320 ohm-cm. And in the temperature field from a room temperature to 300 degrees C, it is also the big description that the change to the resistance of 25 degrees C has the outstanding resistance stability of triple or less figures so that clearly [ this resistor ] from the example mentioned later. Moreover, it has the resistance which does not change at least -100 degrees C to a room temperature.

[0012] Although it is the point of the ease of the manufacture although the process is not exceptionally limited as an approach of manufacturing the ceramic resistor of this invention as long as the above-mentioned configuration is satisfied, and especially vapor growth is desirable and is specifically formed by chemistry vapor phase synthetic methods (CVD method), such as physical vapor phase synthetic methods (PVD), such as sputtering and ion plating, and plasma CVD, Light CVD, MO(Metal-organic) CVD, a CVD method is good also in these. according to these forming-membranes methods -- the [ periodic table ] -- the [ which can compound the aluminum nitride which made 4b group element dissolve superfluously, and is adopted by this invention / periodic table ] -- the ceramic resistor from which 0.01-30 atom % content of 4b group element was done, and the lattice constant of an aluminum nitride crystal changed can be obtained.

[0013] the [ periodic table ] -- Si being chosen as a 4b group element, and as a concrete process using a CVD method It is N<sub>2</sub> as material gas. Gas and NH<sub>3</sub> Gas and SiCl<sub>4</sub> And AlCl<sub>3</sub> Gas is used. Flow rate of these gas can be set to N<sub>2</sub>/AlCl<sub>3</sub> =5-70, SiCl<sub>4</sub>/NH<sub>3</sub> =0.001-3, and NH<sub>3</sub>/AlCl<sub>3</sub> =0.1-10, and membrane formation temperature can be produced 850 degrees C or more by setting up more highly comparatively. SiCl<sub>4</sub> instead of -- SiHCl<sub>3</sub>, SiH<sub>2</sub> Cl<sub>2</sub>, SiH<sub>4</sub>, and Si two H<sub>6</sub> etc. -- you may use -- again -- AlCl<sub>3</sub> Organic aluminum, such as halogenides, such as AlBr, and trimethylaluminum, may be used instead.

[0014] As a base which forms the film on the other hand, although all things can be used, aluminum 2O<sub>3</sub>, AlON, Si<sub>3</sub> N<sub>4</sub>, a diamond, a mullite, ZrO<sub>2</sub>, W, Mo, Mo-Mn, TiN, SiC and WC, carbon, and Si semiconductor material (n

mold or p mold) are also specifically mentioned, but when the sintered compact which makes aluminum nitride a subject also in these takes adhesion into consideration, it is the most desirable.

[0015]

[Function] usually -- although aluminum nitride is a high insulator exceeding volume resistivity  $10^{14}$  ohm-cm -- under the aluminum nitride crystal -- the [ periodic table ] -- 4b group element is dissolved -- making -- aluminum or nitrogen -- the [ periodic table ] -- if 4b group element is made to permute, it will be thought that it makes with the operation which contributes to conductivity as a donor or an acceptor, and raises the conductivity of a crystal. the [ moreover, / to an aluminum nitride crystal / periodic table ] -- dissolution of 4b group element can be judged by change of a lattice constant. the [ for example, / periodic table ] -- although the lattice constant of the aluminum nitride which does not contain 4b group element was 4.994Å in 3.120Å and a c-axis at the a-axis -- the [ periodic table ] -- 4b group element dissolves -- it is alike, and it follows and an a-axis and a c-axis change. And if a lattice constant is made into the value which shifted from these values to 0.003–0.030Å by the a-axis, and was shifted to the large value or the small value only 0.004–0.080Å by the c-axis, volume resistivity is controllable to  $10^{13}$  or less ohm-cm.

[0016] And the ceramic resistor of this invention has the small resistance change to temperature, for example, when it is general aluminum nitride, to changing from  $10^{16}$  ohm-cm to  $10^{11}$  ohm-cm, by the ceramic resistor of this invention, has the description that only triple or less figures change for example, from  $10^{13}$  ohm-cm even with  $10^{11}$  ohm-cm, and maintains a volume resistivity value with the small rate of change to  $-100$ –degree C low temperature in the temperature requirement from a room temperature (25 degrees C) to 300 degrees C.

[0017] Therefore, usefulness is [ as opposed to / especially / applications, such as an electrostatic chuck in the semiconductor fabrication machines and equipment for which the resistance stabilized over the large temperature requirement is needed, ] high.

[0018]

[Example]

The AlN film was formed in the base front face which consists of a nature sintered compact of example 1 aluminum nitride with the chemistry vapor phase synthetic method. Membrane formation of the AlN film put the base into the furnace heated at 900 degrees C by the outside heat type, nitrogen was poured for 8SLM(s), and it poured ammonia for  $\text{SiCl}_4$  gas of 1SLM and 0–0.5SLM, and set the pressure to 50torr(s). Furthermore, the aluminum chloride ( $\text{AlCl}_3$ ) was introduced by the flow rate of 0.3SLM(s), the reaction was started, and the film of 400–micrometer thickness was formed (sample No.1–9).

[0019] Angle correction was performed by having made Si (SRM640b) into the standard sample with the X-ray diffraction method to the obtained film, and it computed by the peak top method. Measurement indices of crystal plane were (100), (002), (101), (102), (110), (103), (112), and (004). Moreover,  $-100$  degrees C, a room temperature, and 300–degree C volume resistivity were measured, and it was shown in Table 1. Moreover, the  $-100$ –600–degree C volume resistivity of No.5 was shown in drawing 1.

[0020] The AlN film was formed in the base front face which consists of a nature sintered compact of example 2 aluminum nitride with the chemistry vapor phase synthetic method. It puts into the furnace which heated the base at 900 degrees C by the outside heat type, and membrane formation of the AlN film is  $\text{CH}_4$  of 1SLM and 0–0.5SLM,  $\text{GeH}_4$ ,  $\text{SnCl}_4$ , and  $\text{Pb}(\text{CH}_3)_4$  about 8SLM(s) and ammonia in nitrogen. Gas was passed and the pressure was set to 50torr(s). Furthermore, the aluminum chloride ( $\text{AlCl}_3$ ) was introduced by the flow rate of 0.3SLM(s), the reaction was started, and the film of about 400–micrometer thickness was formed (sample No.10–13). While computing the lattice constant from the X-ray diffraction method like the example 1 to the obtained film,  $-100$  degrees C, a room temperature, and the volume resistivity in 300 degrees C were measured, and the result was shown in Table 1.

[0021]

[Table 1]

試料 No.	ガス種 (SCCM)	4b族元素 含有量 (atm%)	格子定数 (Å)		体積固有抵抗 (Ω・cm)			
			a 軸	c 軸	-100℃	室温 25℃	300℃	
* 1	SiH <sub>4</sub>	0	0.0001	0	0	$4.5 \times 10^{16}$	$9.0 \times 10^{15}$	$1.2 \times 10^{11}$
2	"	10	0.006	0.004	0.005	$9.8 \times 10^{12}$	$7.2 \times 10^{12}$	$3.5 \times 10^{12}$
3	"	50	0.1	0.008	0.013	$5.1 \times 10^{10}$	$3.0 \times 10^{10}$	$2.5 \times 10^{10}$
4	SiCl <sub>4</sub>	50	0.8	0.005	0.008	$7.4 \times 10^{12}$	$5.5 \times 10^{12}$	$7.3 \times 10^{12}$
5	"	100	3.5	0.007	0.015	$1.5 \times 10^{12}$	$6.4 \times 10^{11}$	$7.8 \times 10^{10}$
6	"	200	7.8	0.010	0.022	$3.5 \times 10^{10}$	$2.2 \times 10^{10}$	$6.9 \times 10^9$
7	"	500	26.7	0.027	0.070	$1.6 \times 10^9$	$2.5 \times 10^8$	$9.7 \times 10^4$
8	"	600	29.0	0.030	0.078	$4.3 \times 10^8$	$3.2 \times 10^8$	$8.0 \times 10^1$
* 9	"	1000	34.9	0.038	0.090	$7.0 \times 10^{14}$	$2.1 \times 10^{14}$	$7.5 \times 10^{10}$
10	CH <sub>4</sub>	100	0.03	0.006	0.012	$5.4 \times 10^{11}$	$5.0 \times 10^{11}$	$4.9 \times 10^{11}$
11	"	500	0.2	0.015	0.049	$8.6 \times 10^7$	$9.5 \times 10^7$	$7.2 \times 10^7$
12	GeH <sub>4</sub>	20	0.05	0.008	0.014	$3.3 \times 10^{11}$	$2.5 \times 10^{11}$	$6.6 \times 10^{10}$
13	SnCl <sub>4</sub>	100	2.0	0.016	0.035	$1.0 \times 10^{10}$	$8.9 \times 10^9$	$9.0 \times 10^9$
14	Pb(CH <sub>3</sub> ) <sub>4</sub>	10	0.1	0.007	0.016	$5.9 \times 10^{11}$	$3.0 \times 10^{11}$	$2.8 \times 10^{11}$

\* 印は本発明の範囲外の試料を示す。

[0022] Si atomic weight and the lattice constant in aluminium nitride are SiCl<sub>4</sub> so that clearly from the result of sample No.1-9 of Table 1. It changes with flow rates. SiCl<sub>4</sub> Do not introduce at all, but in 0.0001 atom % of impurity level, volume resistivity was  $9 \times 10^{15}$  ohm-cm and high insulation, and Si atomic weight SiCl<sub>4</sub> Although the lattice constant also became small gradually while it followed on making a flow rate increase gradually and Si atomic weight in the film increased, the film of sample No.9 was what makes a silicon nitride phase the main phase. In addition, the obtained aluminium nitride film was AlN film which carries out orientation to (002) from X diffraction measurement. However, the silicon nitride crystal phase exists in transmission electron microscope observation, and the amount is SiCl<sub>4</sub>. A flow rate and correlation were found.

[0023] the [ moreover, / other than Si / periodic table ] -- 4b group element -- the [ in aluminium nitride / periodic table ] -- 4b group element atomic weight and a lattice constant -- the [ periodic table ] -- the flow rate of the addition gas containing 4b group element -- changing -- the [ periodic table ] -- the flow rate of the addition gas containing 4b group element is increased gradually -- making -- following -- the [ in the film / periodic table ] -- while the amount of 4b group elements increased, the lattice constant also changed gradually and volume resistivity fell. In addition, the obtained aluminium nitride film is AlN film which carries out orientation to (002) from X diffraction measurement, and that in which a nitride crystal phase exists and is also existed.

[0024]  
[Effect of the Invention] according to [ as explained in full detail above ] this invention -- the [ in aluminium nitride / periodic table ] -- by controlling the amount of 4b group elements, and a lattice constant, the volume resistivity in a room temperature is  $10^{13}$  or less ohm-cm, and the small resistor of a temperature change can be obtained. Therefore, resistance can be changed, without losing the property of aluminium nitride, for example, corrosion resistance.

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TECHNICAL FIELD

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[Industrial Application] This invention relates to the ceramic resistor which makes a subject the aluminum nitride suitable for a heater ingredient, the electrification removal ingredient in thermionic-tube enclosure tubing or semiconductor fabrication machines and equipment, the arm for wafer conveyance, the fixture for wafer handling, etc.

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PRIOR ART

[Description of the Prior Art] Conventionally, generally as an approach for adjusting the electric resistance of the insulating ceramics, adding a conductive ingredient and controlling resistance is performed to the insulating ceramics. For example, adding titanium nitride to an alumina and making electric resistance small is performed. [0003] On the other hand, alumimium nitride is one sort of the non-oxidizing quality ceramics, the application as a structural material or high temperature materials is expected, and having the endurance which was excellent in recently also to the plasma-proof is reported. Therefore, the application as components in semiconductor fabrication machines and equipment, such as an electrostatic chuck, is taken into consideration in this alumimium nitride. However, it is this alumimium nitride itself and a high insulating material, and since it has the resistance of 10<sup>16</sup> or more ohm-cm also at a room temperature, the present condition is having not resulted in utilization.

[0004] The attempt which makes electric resistance small is performed also to such alumimium nitride. For example, adding conductive ingredients, such as aluminum, also to the insulating ceramics of alumimium nitride or boron nitride, and adjusting specific resistance is proposed by JP,56-4509,A. Moreover, in the thin film-like ceramics, making alumimium nitride distribute metal aluminum for example, and obtaining a thin film resistor with a small temperature coefficient of resistance is also proposed by JP,55-50364,B.

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EFFECT OF THE INVENTION

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**TECHNICAL PROBLEM**

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[Problem(s) to be Solved by the Invention] Generally, although the volume resistivity value of an insulator tends to fall with temperature, in the case of alumimium nitride, it is in the inclination which decreases to 1011 or less ohm-cm by 300 degrees C from 1016 ohm-cm at a room temperature, for example. Therefore, since the actuation by which resistance was changed and stabilized was not obtained when using it from a room temperature to a 300-degree C elevated temperature, there were problems, like service temperature conditions have a limit.

[0006] Moreover, in the approach of controlling electric resistance, there were problems -- the property which the insulating ceramics originally has with the property of the conductive ingredient itself is spoiled -- by adding a conductive ingredient. For example, corrosion resistance and endurance were missing and the property of alumimium nitride deteriorated.

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MEANS

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[Means for Solving the Problem] Especially for this invention person etc., electric resistance is a ceramic resistor of 1013 or less ohm-cm to the above-mentioned trouble. 0.005-30 atom % content of 4b group element group's element is carried out. the inside of the insulator which uses as a principal component the alumimium nitride formed by the chemistry vapor phase synthetic method as a result of repeating examination from a viewpoint of the presentation and an organization -- the [ periodic table ] -- By making the element dissolve during an alumimium nitride crystal, and controlling the lattice constant of alumimium nitride in the specific range The volume resistivity of an insulating layer could adjust to the range of 1013 or less ohm-cm, and it found out that the material property by which the temperature change was stabilized in the small large temperature region was acquired, and resulted in this invention.

[0008] namely, the ceramic resistor to which the ceramic resistor of this invention makes an alumimium nitride crystal phase a subject -- it is -- the inside of this resistor -- the [ periodic table ] -- 0.005-30 atom % existence, it carries out, and 4b group element is characterized by the volume resistivity in 25 degrees C being 1013 or less ohm-cm, while the lattice constant in said crystal phase is in the value shifted only 0.004-0.080A by 0.003-0.030A and the c-axis by the a-axis.

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[0011] The ceramic resistor of this invention has the volume resistivity of 1013 or less ohm-cm in 25 degrees C by the above-mentioned configuration, and the lower limit is about 320 ohm-cm. And in the temperature field from a room temperature to 300 degrees C, it is also the big description that the change to the resistance of 25 degrees C has the outstanding resistance stability of triple or less figures so that clearly [ this resistor ] from the example mentioned later. Moreover, it has the resistance which does not change at least -100 degrees C to a room temperature.

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as an approach of manufacturing ceramic resistor of this invention as long as the above-mentioned configuration is satisfied, and especially vapor growth is desirable and is specifically formed by chemistry vapor phase synthetic methods (CVD method), such as physical vapor phase synthetic methods (PVD), such as sputtering and ion plating, and plasma CVD, Light CVD, MO(Metal-organic) CVD, a CVD method is good also in these. according to these forming-membranes methods -- the [ periodic table ] -- the [ which can compound the aluminum nitride which made 4b group element dissolve superfluously, and is adopted by this invention / periodic table ] -- the ceramic resistor from which 0.01-30 atom % content of 4b group element was done, and the lattice constant of an aluminum nitride crystal changed can be obtained.

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## OPERATION

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[Function] usually -- although aluminum nitride is a high insulator exceeding volume resistivity 10<sup>14</sup> ohm-cm -- under the aluminum nitride crystal -- the [ periodic table ] -- 4b group element is dissolved -- making -- aluminum or nitrogen -- the [ periodic table ] -- if 4b group element is made to permute, it will be thought that it makes with the operation which contributes to conductivity as a donor or an acceptor, and raises the conductivity of a crystal. the [ moreover, / to an aluminum nitride crystal / periodic table ] -- dissolution of 4b group element can be judged by change of a lattice constant. the [ for example, / periodic table ] -- although the lattice constant of the aluminum nitride which does not contain 4b group element was 4.994Å in 3.120Å and a c-axis at the a-axis -- the [ periodic table ] -- 4b group element dissolves -- it is alike, and it follows and an a-axis and a c-axis change. And if a lattice constant is made into the value which shifted from these values to 0.003-0.030Å by the a-axis, and was shifted to the large value or the small value only 0.004-0.080Å by the c-axis, volume resistivity is controllable to 10<sup>13</sup> or less ohm-cm.

[0016] And the ceramic resistor of this invention has the small resistance change to temperature, for example, when it is general aluminum nitride, to changing from 10<sup>16</sup> ohm-cm to 10<sup>11</sup> ohm-cm, by the ceramic resistor of this invention, has the description that only triple or less figures change for example, from 10<sup>13</sup> ohm-cm even with 10<sup>11</sup> ohm-cm, and maintains a volume resistivity value with the small rate of change to -100-degree C low temperature in the temperature requirement from a room temperature (25 degrees C) to 300 degrees C.

[0017] Therefore, usefulness is [ as opposed to / especially / applications, such as an electrostatic chuck in the semiconductor fabrication machines and equipment for which the resistance stabilized over the large temperature requirement is needed, ] high.

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[Translation done.]

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EXAMPLE

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## [Example]

The AlN film was formed in the base front face which consists of a nature sintered compact of example 1 aluminum nitride with the chemistry vapor phase synthetic method. Membrane formation of the AlN film put the base into the furnace heated at 900 degrees C by the outside heat type, nitrogen was poured for 8SLM(s), and it poured ammonia for SiCl<sub>4</sub> gas of 1SLM and 0-0.5SLM, and set the pressure to 50torr(s). Furthermore, the aluminum chloride (AlCl<sub>3</sub>) was introduced by the flow rate of 0.3SLM(s), the reaction was started, and the film of 400-micrometer thickness was formed (sample No.1-9).

[0019] Angle correction was performed by having made Si (SRM640b) into the standard sample with the X-ray diffraction method to the obtained film, and it computed by the peak top method. Measurement indices of crystal plane were (100), (002), (101), (102), (110), (103), (112), and (004). Moreover, -100 degrees C, a room temperature, and 300-degree C volume resistivity were measured, and it was shown in Table 1. Moreover, the -100-600-degree C volume resistivity of No.5 was shown in drawing 1.

[0020] The AlN film was formed in the base front face which consists of a nature sintered compact of example 2 aluminum nitride with the chemistry vapor phase synthetic method. It puts into the furnace which heated the base at 900 degrees C by the outside heat type, and membrane formation of the AlN film is CH<sub>4</sub> of 1SLM and 0-0.5SLM, GeH<sub>4</sub>, SnCl<sub>4</sub>, and Pb (CH<sub>3</sub>)<sub>4</sub> about 8SLM(s) and ammonia in nitrogen. Gas was passed and the pressure was set to 50torr(s). Furthermore, the aluminum chloride (AlCl<sub>3</sub>) was introduced by the flow rate of 0.3SLM(s), the reaction was started, and the film of about 400-micrometer thickness was formed (sample No.10-13). While computing the lattice constant from the X-ray diffraction method like the example 1 to the obtained film, -100 degrees C, a room temperature, and the volume resistivity in 300 degrees C were measured, and the result was shown in Table 1.

[0021]

[Table 1]

試料 No.	ガス種 (SCCM)	4b族元素 含有量 (atm%)	格子定数 (Å)		体積固有抵抗 (Ω-cm)			
			a 軸	c 軸	-100℃	室温 25℃	300℃	
* 1	SiH <sub>4</sub>	0	0.0001	0	0	$4.5 \times 10^{16}$	$9.0 \times 10^{15}$	$1.2 \times 10^{11}$
2	"	10	0.006	0.004	0.005	$9.8 \times 10^{12}$	$7.2 \times 10^{12}$	$3.5 \times 10^{12}$
3	"	50	0.1	0.008	0.013	$5.1 \times 10^{10}$	$3.0 \times 10^{10}$	$2.5 \times 10^{10}$
4	SiCl <sub>4</sub>	50	0.8	0.005	0.008	$7.4 \times 10^{12}$	$5.5 \times 10^{12}$	$7.3 \times 10^{12}$
5	"	100	3.5	0.007	0.015	$1.5 \times 10^{12}$	$6.4 \times 10^{11}$	$7.8 \times 10^{10}$
6	"	200	7.6	0.010	0.022	$3.5 \times 10^{10}$	$2.2 \times 10^{10}$	$6.9 \times 10^8$
7	"	500	26.7	0.027	0.070	$1.6 \times 10^8$	$2.5 \times 10^8$	$9.7 \times 10^4$
8	"	600	29.0	0.030	0.078	$4.3 \times 10^7$	$3.2 \times 10^8$	$8.0 \times 10^3$
* 9	"	1000	34.9	0.038	0.090	$7.0 \times 10^{14}$	$2.1 \times 10^{14}$	$7.5 \times 10^{10}$
10	CH <sub>4</sub>	100	0.03	0.006	0.012	$5.4 \times 10^{11}$	$5.0 \times 10^{11}$	$4.9 \times 10^{11}$
11	"	500	0.2	0.015	0.049	$8.6 \times 10^7$	$9.5 \times 10^7$	$7.2 \times 10^7$
12	GeH <sub>4</sub>	20	0.05	0.008	0.014	$3.3 \times 10^{11}$	$2.5 \times 10^{11}$	$6.6 \times 10^{10}$
13	SnCl <sub>4</sub>	100	2.0	0.016	0.035	$1.0 \times 10^{10}$	$8.9 \times 10^9$	$9.0 \times 10^9$
14	Pb(CH <sub>3</sub> ) <sub>4</sub>	10	0.1	0.007	0.016	$5.9 \times 10^{11}$	$3.0 \times 10^{11}$	$2.8 \times 10^{11}$

\* 印は本発明の範囲外の試料を示す。

[0022] Si atomic weight and the lattice constant in aluminum nitride are SiCl<sub>4</sub> so that clearly from the result of sample No.1-9 of Table 1. It changes with flow rates. SiCl<sub>4</sub> Do not introduce at all, but in 0.0001 atom % of impurity level, volume resistivity was  $9 \times 10^{15}$  ohm-cm and high insulation, and Si atomic weight SiCl<sub>4</sub> Although the lattice constant also became small gradually while it followed on making a flow rate increase gradually and Si atomic weight in the film increased, the film of sample No.9 was what makes a silicon nitride phase the main phase. In addition, the obtained aluminum nitride film was AlN film which carries out orientation to (002) from X diffraction measurement. However, the silicon nitride crystal phase exists in transmission electron microscope observation, and the amount is SiCl<sub>4</sub>. A flow rate and correlation were found.

[0023] the [ moreover, / other than Si / periodic table ] -- 4b group element -- the [ in aluminum nitride / periodic table ] -- 4b group element atomic weight and a lattice constant -- the [ periodic table ] -- the flow rate of the addition gas containing 4b group element -- changing -- the [ periodic table ] -- the flow rate of the addition gas containing 4b group element is increased gradually -- making -- following -- the [ in the film / periodic table ] -- while the amount of 4b group elements increased, the lattice constant also changed gradually and volume resistivity fell. In addition, the obtained aluminum nitride film is AlN film which carries out orientation to (002) from X diffraction measurement, and that in which a nitride crystal phase exists and is also existed.

[Translation done.]



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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing showing change of the -100 degrees C - 600 degrees C volume resistivity of sample No.5.

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[Translation done.]

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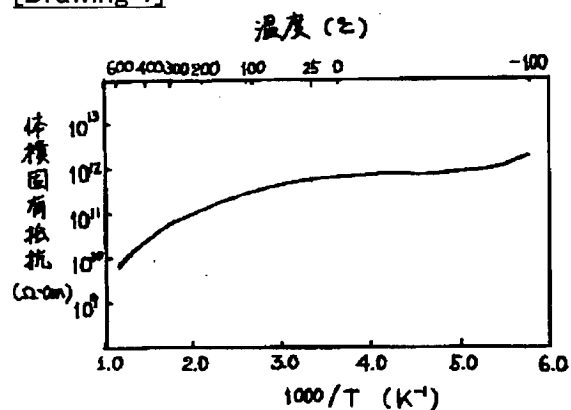
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DRAWINGS

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[Drawing 1]



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[Translation done.]